NASA
Technical Memorandum 82817

AVRADCOM
Technical Report 82-C-2

Thermal Degradation of the Tensile Properties of Unidirectionally Reinforced FP-Al_2O_3/EZ 33 Magnesium Composites

R. T. Bhatt
Propulsion Laboratory
AVRADCOM Research and Technology Laboratories
Lewis Research Center
Cleveland, Ohio

and

H. H. Grimes
Lewis Research Center
Cleveland, Ohio

Prepared for the
One-hundred-eleventh Annual Meeting of the
American Institute of Mining,
Metallurgical and Petroleum Engineers
Dallas, Texas, February 14–18, 1982

NASA

U.S. ARMY AVRADCOM

THERMAL DEGRADATION OF THE TENSILE PROPERTIES OF UNDIRECTIONALLY REINFORCED FP-Al_2O_3/EZ 33 MAGNESIUM COMPOSITES

R. T. Bhatt
Propulsion Laboratory
AVRADCOM Research and Technology Laboratories
Lewis Research Center
Cleveland, Ohio 44135

and

H. H. Grimes
National Aeronautics and Space Administration
Lewis Research Center
Cleveland, Ohio 44135

Abstract

The effects of isothermal and cyclic exposure on the room temperature axial and transverse tensile strength and dynamic flexural modulus of 35 and 55 volume percent FP-Al_2O_3/EZ 33 magnesium composites have been studied. The composite specimens were continuously heated in a sand bath maintained at 350° C for up to 150 hours or thermally cycled between 50° and 250° C or 50° and 350° C for up to 3000 cycles. Each thermal cycle lasted for a total of six minutes with a hold time of two minutes at the maximum temperature. Results indicate no significant loss in the room temperature axial tensile strength and dynamic flexural modulus of composites thermally cycled between 50° and 250° C or of composites isothermally heated at 350° C for up to 150 hours from the strength and modulus data for the untreated, as-fabricated composites. In contrast, thermal cycling

between 50° and 350° C caused considerable loss in both room temperature strength and modulus. Fractographic analysis and measurement of composite transverse strength and matrix hardness of thermally cycled and isothermally heated composites indicated matrix softening and fiber/matrix debonding due to void growth at the interface and matrix cracking as the likely causes of the strength and modulus loss behavior.

Introduction

Composites of magnesium alloys reinforced with FP-Alumina are being considered for aerospace applications because of their high specific strength and modulus, and because of their relative ease of fabrication. Some of these applications require repeated high temperature exposure of the composite material for extended periods of time. In a recent study, Bhatt, et al.,[1] measured the room temperature axial and transverse strengths and moduli of three different, unidirectional FP-Al_2O_3/magnesium composites prepared by Dupont by a liquid metal infiltration technique. The matrix materials used were EZ 33 (2 to 4 percent rare earths: 2 to 3 percent Zn: 0.5 to 1 percent Zr: bal Mg), QH 21A (2 to 3 percent Ag, 0.6 to 1.6 percent Th: 0.6 to 1.5 percent rare earths; 0.4 to 1 percent Zr: bal Mg) and pure magnesium. The effect of short term (120 hr) isothermal exposure at 350° and 425° C on the above composite properties was also determined. At these temperatures all these composites showed both axial and transverse strength degradation. The major cause of this degradation was matrix softening.

The isothermal exposure temperatures used in this earlier study were chosen to insure strength degradation and therefore represent more severe temperature condition than would be experienced by the composite in normal use. On the other hand, the cyclic environments the composites will experience in normal use may represent a severe degradation condition even at the lower temperatures. This is because under cyclic heating conditions the plastic deformation which may occur in the matrix due to thermally induced stresses will be at least in part irreversible leading to cumulative effects such as matrix cracking or void growth.

The objective of this investigation, therefore, was to characterize more fully the useful range of application of a selected FP-Al_2O_3/magnesium composite, as determined by tensile strength and modulus measurements after cyclic thermal exposure.

Of the three magnesium matrix alloys previously studied, EZ 33 was choosen for this investigation because it bonded well to the FP-Alumina fiber resulting in a composite which displayed adequate axial and transverse strengths combined with an excellent modulus. While the properties determined in that study for the FP-Al_2O_3/QH 21A composites were generally similar, this material was found to be poorly bonded so that the full advantage of the stronger matrix alloy was not realized. It was felt that the poorly bonded composite would not perform well in cyclic testing.

In this study, 35 and 55 volume percent fiber unidirectionally aligned composites were exposed in air to temperatures of either 250° or 350° C for up to 3000 cycles. Isothermal exposure at 350° C was used as a baseline to evaluate the additional effect of cycling. Metallographic and fractog-

raphic analysis and matrix hardness measurements were also conducted on untreated and thermally cycled composite material in an attempt toward understanding of the failure mechanisms involved.

Experimental

The FP-Al_2O_3/EZ 33 Mg composites used in this study were fabricated by the Dupont Pioneering Research Laboratory using molten metal infiltration techniques. The nominal FP-Al_2O_3 fiber content used was either 35 or 55 volume percent. The fibers were aligned unidirectionally in a EZ 33 magnesium alloy matrix having the composition previously given. Plates of 0.25 cm thickness were cast and cut into specimens 12.7 cm long and 1.25 cm wide. The fiber orientation was either parallel to the specimen length (0°) for axial testing or perpendicular to the specimen length (90°) for transverse testing. Thermal cycling was done by alternately dipping a frame supporting six specimens into a hot (250° or 350° C) fluidized sand bath and then into a cold bath that equilibrated near 50° C. Each complete thermal cycle lasted for six minutes. Typical time-temperature profiles of the composite specimen cycled to 250° or 350° C are shown in Fig. 1. The time at temperature during each cycle was approximately two minutes.

Similar composite specimens were also isothermally heated at 350° C in a sand bath for periods up to 150 hours, which corresponds to a time about 50 percent longer than the time at temperature for 3000 cycle experiments.

After cycling to a predetermined number of cycles or isothermal heating to set time periods, the specimens were removed from the bath and aluminum doublers were adhesively bonded to the specimen ends.

Tensile testing was done in an Instron testing machine equipped with wedge type grips. The specimens were pulled to failure at a constant cross head speed of 0.126 cm/min. Matrix hardness was measured in a Vickers hardness testing machine using a diamond pyramid indentor and a 25 gm load.

A flexural modulus test, similar to that of McDanels et. al[2], was used for measuring the dynamic modulus of the composite specimen. In this test, the composite specimen is supported on a pair of steel wires. The distance between the wires was set to 0.7758L, where L is the specimen length. This corresponds to the two nodal positions for the flexural deformation of a free bar of the given dimensions in flexural vibration. The specimen is driven by a piezo-electric transducer positioned at one end of the specimen. A similar transducer placed at the other end of the specimen was used as a detector. The block diagram of the transducer drive and detection system used is shown in Fig. 2.

For determination of the dynamic flexural modulus, E, the variable drive transducer was manually tuned to that frequency which produced resonance (the maximum flexural displacement) in the specimen. For the free-free flexural modes of rectangular bar specimens with low damping, the resonant frequency, ω, is related to E by the equation:

$$E = \frac{12 \times (2\pi \times \omega)^2}{b^2} \left(\frac{M}{W}\right)\left(\frac{l}{h}\right)^3 \qquad (1)$$

where b is a constant and M, h, W and l are the specimen mass, thickness, width and length respective. For $l/h > 100$, $b = 22.37$. For $l/h < 100$, $b < 22.37$. The exact value of b can be determined from specimen dimensions.[3] Thus, by the measurement of the resonant frequency ω, and the dimensions and weight of the specimen, the dynamic flexural modulus of the specimen can be calculated from Eq. (1).

Results and Discussions

The room temperature flexural moduli of the 35 and 55 fiber volume percent FP-Al_2O_3/EZ 33 Mg composites cycled to 250° or 350° C to a maximum of 3000 cycles are shown in Fig. 3. The flexural moduli of untreated composites are also shown in Fig. 3 for base line comparison. The data points represent the range and average value for at least three determinations. The modulus values for 35 and 55 fiber volume composites thermally cycled to 250° C show no significant change from the modulus values of as-fabricated unheated composites. However both composites, thermally cycled to 350° C show a loss in flexural modulus proportional to the number of thermal cycles. After 3000 cycles to 350° C, both the 35 and 55 fiber volume composites degraded to near 80 percent of the modulus values of unheated composite specimens. Also, shown in Fig. 3 is the modulus values for similar composite specimens isothermally treated at 350° C for 150 hours, a time approximately 50 percent longer than the cumulative time at temperature for the 3000 cycle test. These specimens did not show any loss in flexural modulus even after 150 hours of exposure. In the cyclic tests, while the specimen length and weight remained nearly the same, the width and thickness of the specimen increased continuously with cycling. In isothermally heated composites, however, no dimensional changes were measured even after 150 hours of exposure at 350° C. The losses in the dynamic modulus of the cycled composites were also found to be proportional to the width and thickness changes.

The room temperature axial tensile strengths of the 35 and 55 volume percent composites cycled to 250° or 350° C to a maximum of 3000 cycles are shown in Figs. 4 and 5. Again, the data points indicate the range and the average value for typically three tests. The axial strength data for the 35 and 55 fiber volume percent composites without thermal treatment are also shown in Figs. 4 and 5 respectively for a base line comparison. The strength data, as seen in Fig. 4, for the 35 fiber volume percent composites cycled to 250° C show no appreciable degradation from the untreated composite strength of 0.33 GN/m^2 even after as many as 3000 cycles. However, cycling this composite to 350° C resulted in an initial drop in strength from the base line value of 0.33 GN/m^2 to 0.315 GN/m^2 after only 20 cycles, then no further drop in strength until after 2000 cycles and finally a second loss of strength to 0.285 GN/m^2.

For the 55 fiber volume percent composite data shown in Fig. 5, a somewhat different axial strength degradation behavior was observed. Composite cycled 3000 times to 250° C showed a modest loss of strength from the base line value of 0.50 GN/m^2, to 0.47 GN/m^2. Similar composites cycled to 350° C, on the other hand, showed a rapid loss of strength from the base line value of 0.50 GN/m^2 within the first 20 thermal cycles. Additional cycling resulted in a gradual decrease in strength to 0.33 GN/m^2 after 3000 cycles.

The base line axial strength data and strength data measured after 1000, 2000 and 3000 cycles from Figs. 4 and 5 are replotted in Fig. 6 against maximum cycle temperature to better illustrate the temperature dependence of the strength degradation. Clearly from Fig. 6 no significant strength loss occurred for either the 35 or 55 fiber volume percent composites when cycled to 250° C, even for as many as 3000 cycles. At 350° C, however, a rapid strength loss occurs. For the 55 fiber volume percent composite this loss appears to be more a function of cycle temperature than number of cycles. Also shown in Fig. 6 is the range and average value of all the strength data of similar composite specimens which have been isothermally heated at 350° C for 150 hours a time 50 percent longer than the cumulative time-at-temperature for specimens cycled 3000 times. The 35 fiber volume percent composite specimens isothermally heated at 350° C show strength values similar to the strength values of untreated composites, whereas the 55 fiber volume percent composite specimens show a 10 percent loss of strength to a value of 0.45 GN/m^2. In all cases, however, the strength values of isothermally heated specimens were equal to or higher than the strength values for thermally cycled composite specimen.

The greater degradation observed for cyclically heated composites is seen as evidence of a mechanism involving more than a simple thermally activated process. If a single thermally activated process were operating we would expect similar behavior for specimens which were cyclically heated or isothermally heated for an equivalent time at temperature. A likely candidate for this additional mechanism is one which involves the generation of large matrix stresses due to the differences between the thermal expansions of the fiber and matrix during heating. The maximum thermally induced matrix stress in fiber composites can be obtained from the equation derived by Piggott[4]

$$\sigma_m \simeq \frac{3\,(\alpha_m - \alpha_f)\,(\Delta T)\,V_f E_m E_f}{E_c + E_f\,(1 + \gamma_m V_m) - \gamma_f E_m}$$

where α is the thermal expansion coefficient, V is the fiber volume fraction, E is the elastic modulus, γ is poisson's ratio, and ΔT is the cyclic temperature range. The subscripts m, f, and c refer to the matrix, fiber and composite, respectively. From this study we use; $\Delta T = 200°$ or $300°$ C and measured values of E_c. We also use $\alpha_m = 25.4$ μ/cm/cm/°C, $\alpha_f = 5.7$ μ/cm/cm/°C, $E_f = 379$ GN/m^2 and $E_m = 44.8$ GN/m^2 [5], and $\gamma_f = 0.2$, $\gamma_m = 0.33$ [6,7]. The maximum matrix thermal stresses, σ_m, calculated for 35 and 55 fiber volume percent composites are shown in Table I.

Table I

$(T_2 - T_1) = \Delta T$, °C	σ_m, GN/m^2	
	35 Vol percent	55 Vol percent
200	0.119	0.174
300	.179	.262

These stresses exceed even the room temperature yield stress value of 0.11 GN/m^2 for the EZ 33 magnesium alloy.[7] In the thermally cycled composite, therefore plastic deformation will occur during each cycle. The cumulative effect of such repeated deformation has been observed to produce voids in the matrix in highly constrained regions where the matrix deformation cannot be reversed.[8,9] Evidence of similar void growth and matrix cracking in this composite is seen in Fig. 7 which shows microphotographs of the 55 volume percent FP-Al_2O_3/EZ 33 Mg composite before thermal exposure and after cyclic heating to 350° C. The voids here appear to extend to the fiber/matrix interfaces. The presence of voids or cracks at the interface will result in a loss of fiber/matrix bonding.

Whether the loss of axial strength results from fiber/matrix debonding or from a weakening of the fiber or the matrix cannot be determined from these micrographs. However, some insight may be obtained from the results of transverse strength of the composite and from matrix hardness measurements. Figure 8 shows the transverse strength data for the 35 and 55 fiber volume percent composites thermally cycled to 250° or 350° C. It is obvious from this figure that the transverse strength decreases significantly within the first 20 cycles at both temperatures. Further thermal cycling to a maximum of 3000 cycles resulted in a more gradual loss of strength to values as low as 0.055 GN/m^2 for 55 fiber volume FP-Al_2O_3/EZ 33 Mg composite. The magnitude of the initial loss of strength, as seen in Fig. 8, increases markedly with maximum cycle temperature and with increased fiber volume fraction for the higher temperature tests. Also shown in Fig. 8 are the transverse strength data for similar composites isothermally heated at 350° C from 1 hour to 150 hours. As with the data for cycled composites, a rapid initial loss in strength was observed at 350° C for both 35 and 55 fiber volume composites. However, for these composites no further degradation was measured for exposure up to 150 hours. Thus, again it appears that an additional degradation mechanism is occurring during cycling.

The rapid strength loss in the first 20 cycles or within one hour of isothermal exposure at 350° C, appears to correlate better, however, with hardness measured on the void free regions of matrix before and after heat treatment. The matrix hardness data for the 55 volume percent FP-Al_2O_3/EZ 33 Mg composites cycled to 350° C or isothermally heated at 350° C up to 150 hours are plotted in Fig. 9. Each data point is an average of at least ten measurements. The hardnesses of both thermally cycled and isothermally heated composites decreased to nearly one half of the value for unheated as fabricated material within 20 cycles or within one hour of heating. Further cycling to 3000 cycles or heating to 150 hours resulted in no further hardness decrease. This matrix softening correlates exactly with the transverse strength loss for the isothermally heated composites and is assumed to be responsible for this loss.

Matrix softening, however cannot explain the additional gradual transverse strength loss between 20 and 3000 cycles for thermally cycled specimens. An additional degradation mechanism is indicated for the cycled composites. Further insight into this additional mechanism results from examination of the transverse fracture surfaces of the cycled composites. Typical fracture surfaces of the untreated and thermally cycled composites for 20, 1000 and 2000 cycles are shown in Fig. 10. As previously observed

in Fig. 7 for unfractured specimens, there is an increasing tendency for void growth at the fiber/matrix interface with cycling. Since isothermally heated composites showed no voids even after 150 hours, we associate the gradual strength decrease with the number of cycles as shown in Fig. 8 with the growth of voids.

Matrix softening and void growth can also explain the axial strength and modulus loss behavior. The loss of fiber/matrix bonding caused by void growth at the interface will have greater effect on the axial strength and modulus properties than that due to the matrix softening. Indeed these effects were observed in the axial strength and modulus data of cycled or isothermally heated composites.

Summary

The effects of cyclic and isothermal heat treatments on the axial and transverse tensile strengths and axial moduli of 35 and 55 volume percent FP-Al_2O_3/EZ 33 Mg composites have been evaluated to understand the cause of thermally induced strength degradation and to help determine the limiting use conditions for these composites. Specific findings are as follows:

1. Thermal cycling of FP Al_2O_3/EZ 33 Mg composites to 250° C for 2000 cycles did not cause any appreciable room temperature strength or modulus loss compared with base line data for untreated composites. In contrast composites thermally cycled to 350° C showed considerable loss in both strength and modulus, with the 55 volume percent composites degrading more than the 35 volume percent composites.

1. Measurement of the transverse strength and matrix hardness, and fractographic analysis of thermally cycled composites indicated interface void formation, matrix cracking and matrix softening as prime contributors to observed strength and modulus losses. These results and the observed fiber volume dependence are consistant with a degradation mechanism based on thermal induced stresses in the matrix.

3. No appreciable loss in the axial dynamic modulus of these composites was observed after isothermal exposure. Small strength losses observed in isothermally heated composites were attributed to matrix softening.

4. The high temperature mechanical properties were not measured for the composite in this study. This would be required to properly design using FP-Al_2O_3/EZ 33 composite. However, aside from the usual matrix softening at higher temperatures, this study indicates that we would not expect additional axial strength or modulus degradation resulting from either isothermal or cyclic exposure below 2000 cycles or 250° C.

References

1. R. T. Bhatt and H. H. Grimes: "Modulus, Strength and Thermal Exposure Studies of FP-Al_2O_3/Aluminum and FP-Al_2O_3/Magnesium Composites," paper presented at the 5th Annual Conference on Composite Materials, American Ceramic Society, Cocoa Beach, Fl, Jan., 1981.

2. D. L. McDanels and R. A. Signorelli: NASA TN D-8204, 1976.

3. J. DiCarlo and J. E. Maisel: Conference on Composite Materials; Testing and Design, 5th, pp. 201-227, ASTM STP 674, 1979.

4. M. R. Piggott: Load Bearing Fiber Composites, p. 208, Pergamon Press., Oxford, 1980.

5. A. K. Dhingra and W. H. Krueger: New Engineering Material - Magnesium Castings Reinforced with Dupont Continuous Alumina Fiber FP," paper presented at the International Magnesium Association 36th Annual World Conference on Magnesium, Oslo, Norway, June 25-26, 1979.

6. J. F. Bacon, K. T. Prewo and R. D. Veltri: ICCM/2, Proceedings of the Second International Conference on Composite Materials, B. Noton, R. Signorelli, K. Strect, and L. Phillips, eds., pp. 753-769, Metallurgical Society of AIME, Warrendale, PA, 1978.

7. Metals Handbook, 8th Edition, Vol. I, American Society for Metals, Metals Park, OH, 1961.

8. P. Shahinian: SAMPE Quart., Vol. 2, 1970, pp. 28-35.

9. W. H. Kim, M. J. Koczak and A. Lawley: New Developments and Applications in Composites, D. Kuhlmann - Wilsdorf and W. C. Harrigan, eds., pp. 40-53, Metallurgical Society of AIME, Warrendale, PA, 1979.

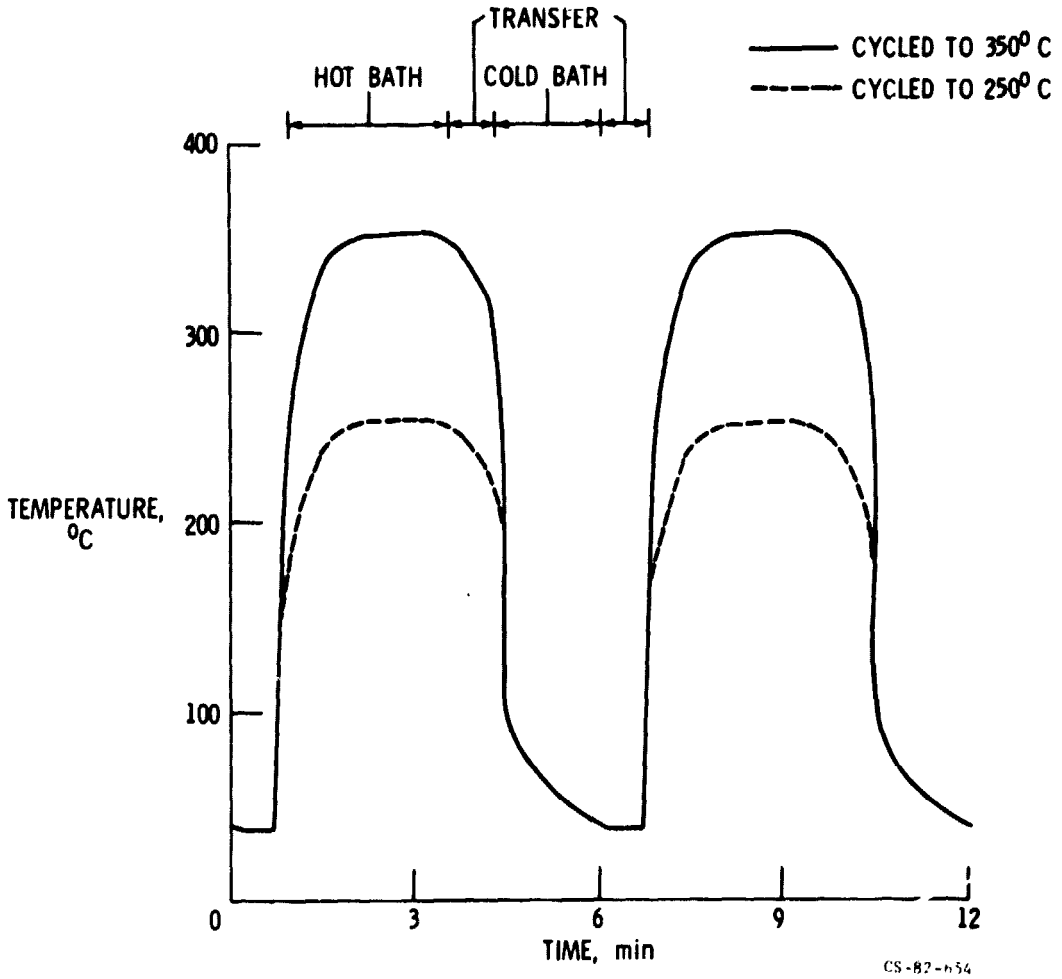


Figure 1. - Typical thermal cycle temperature-time profiles.

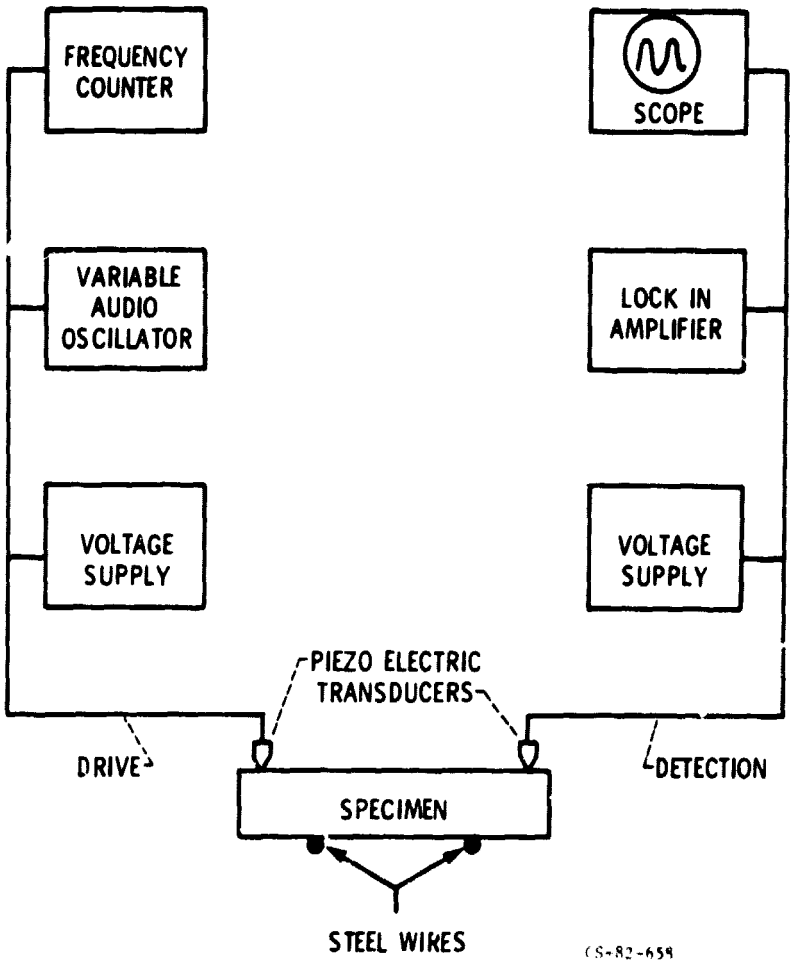


Figure 2. - Block diagram of the piezo electric transducer drive and detection system.

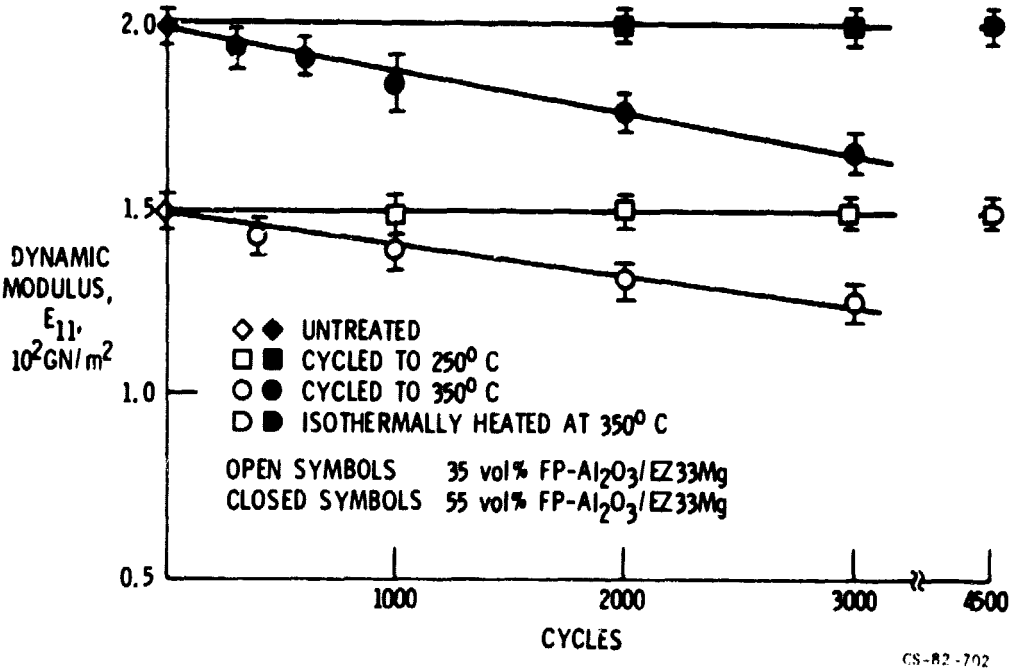


Figure 3. - Room temperature dynamic moduli of FP-Al_2O_3/EZ33Mg composites after cycling to 250° C or 350° C for indicated number of cycles dynamic moduli of composites isothermally heated at 350° C for 150 hours are shown. (Dynamic moduli of untreated composites are shown at zero cycles.)

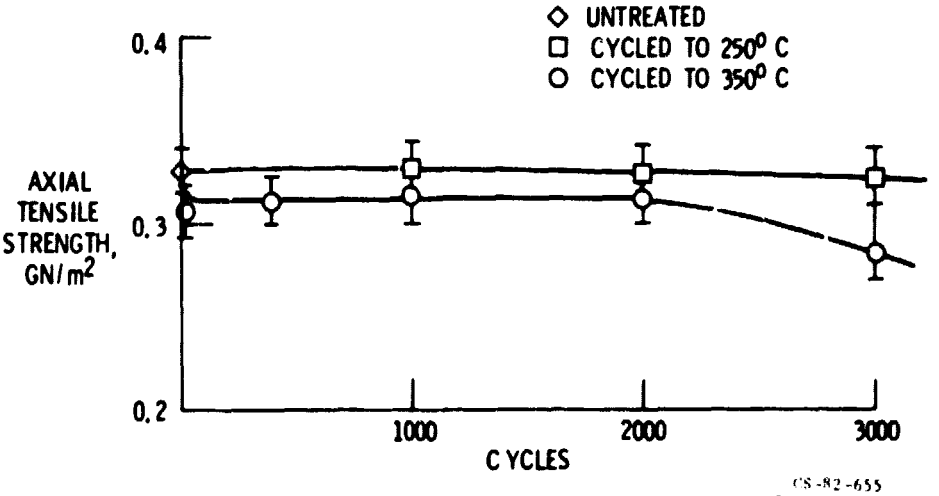


Figure 4. - Room temperature axial tensile strengths of 35 volume percent FP-Al_2O_3/EZ33Mg composites after cycling to 250° C or 350° C for indicated number of cycles strengths of untreated composites are shown at at zero cycles.

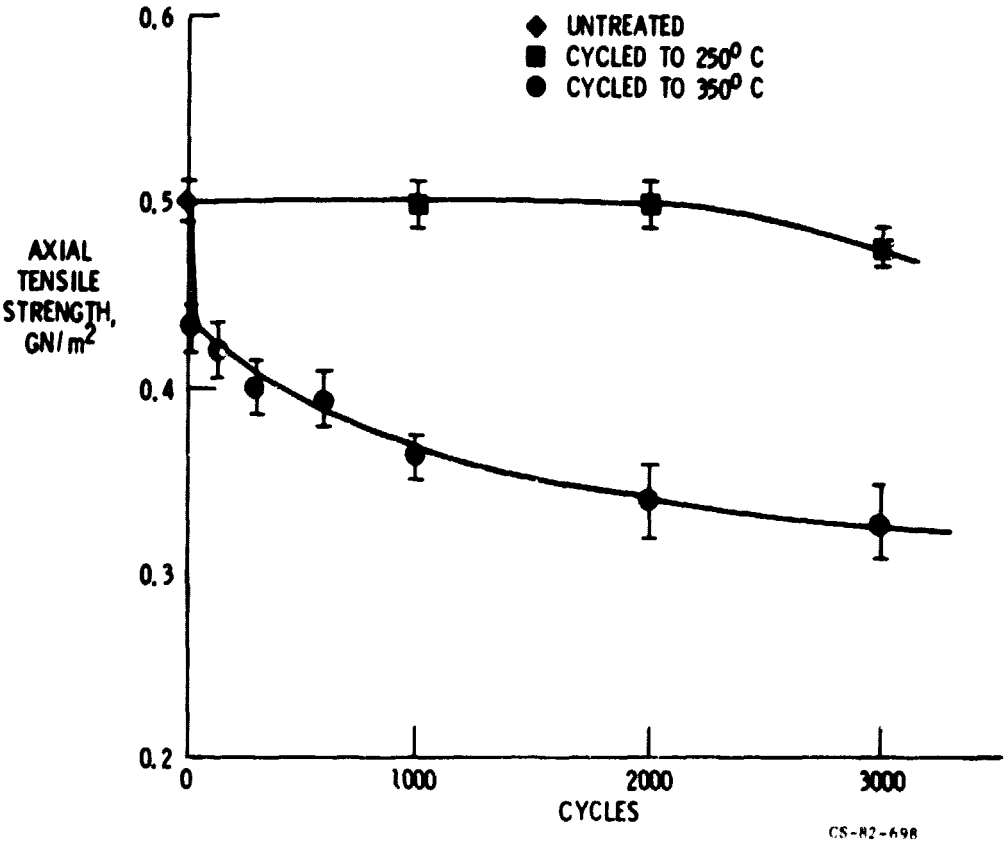


Figure 5. - Room temperature axial tensile strengths of 55 volume percent FP-Al_2O_3/EZ33Mg composites after cycling to 250° C or 350° C for indicated number of cycles. (Strengths of untreated composites are shown at zero cycles.)

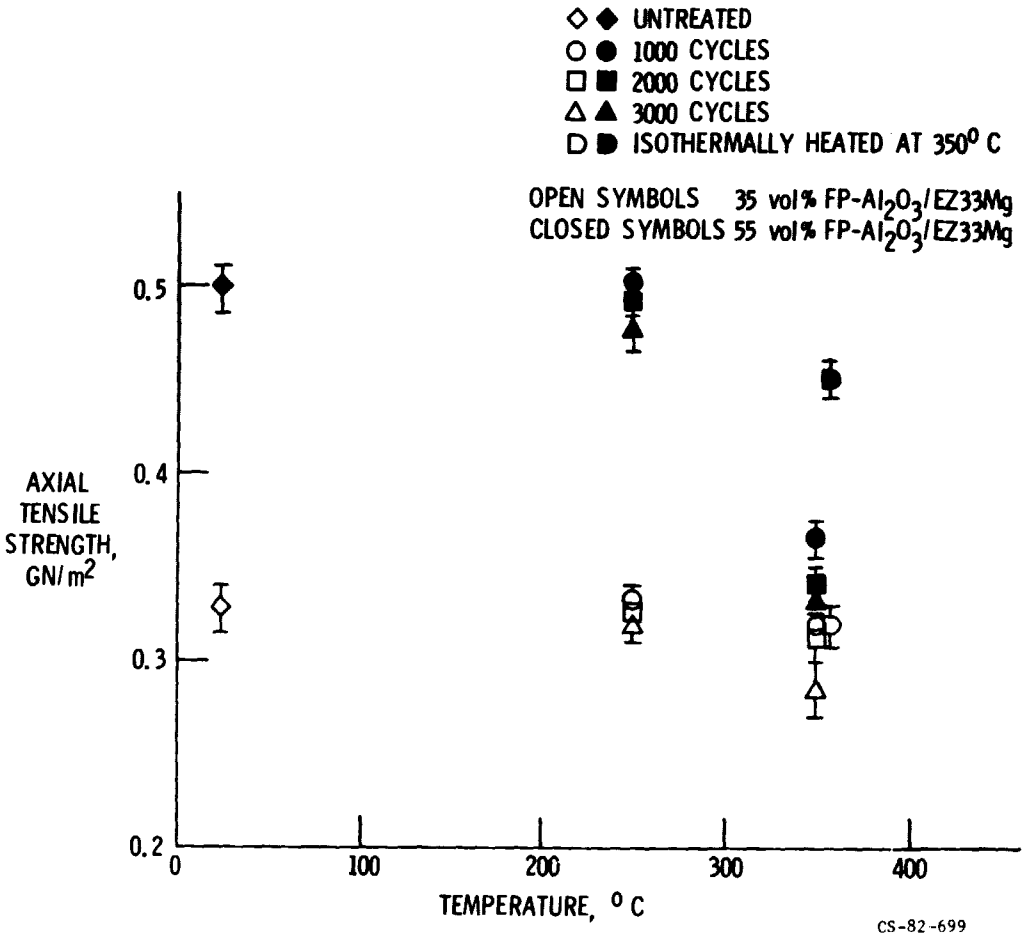
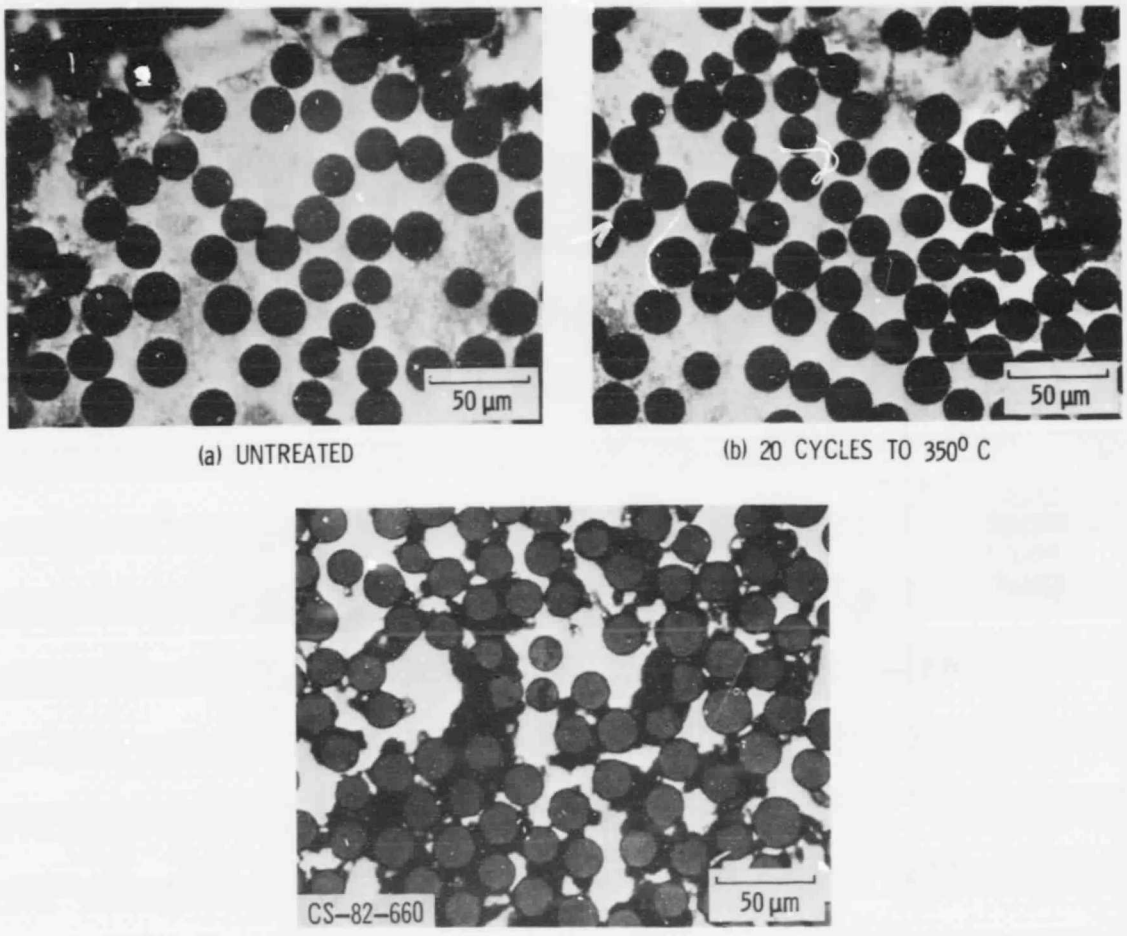


Figure 6. - Room temperature axial tensile strengths of FP-Al_2O_3/EZ33Mg composites cycled 1000, 2000 and 3000 times to indicated temperatures. (Strengths of similar composites isothermally heated at 350° C for 150 hours are also shown.)



(a) UNTREATED

(b) 20 CYCLES TO 350° C

(c) 2000 CYCLES TO 350° C

Figure 7. - Photomicrograph of untreated and thermally cycled composites after indicated number of cycles.

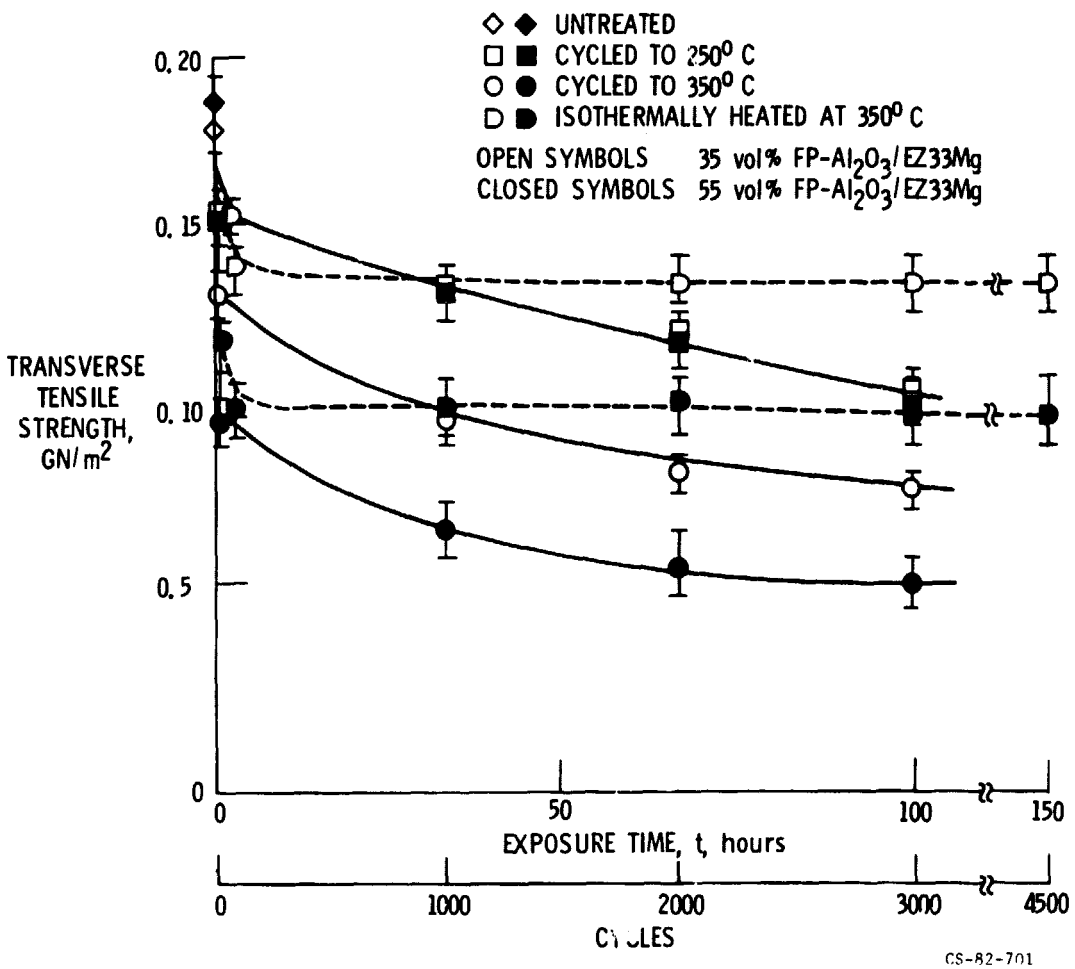


Figure 8. - Room temperature transverse tensile strengths of FP-Al_2O_3/EZ33Mg composites after cycling to 250° C or 350° C for up to 150 hours are shown. (Strengths of untreated composites are shown at zero cycles.)

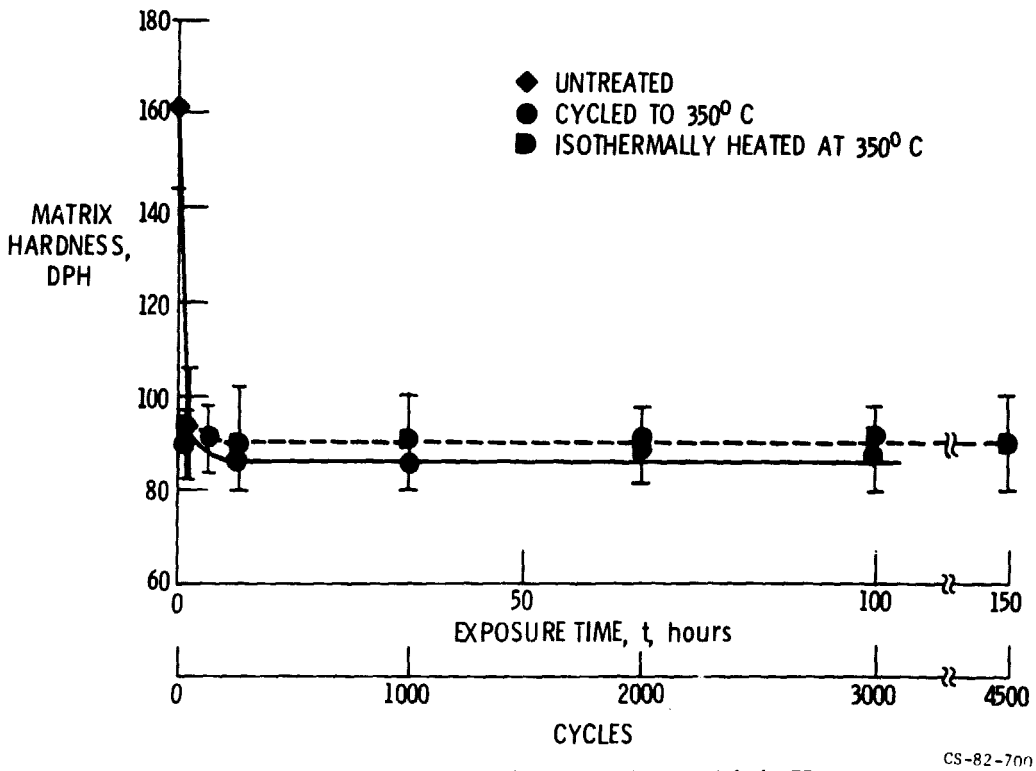
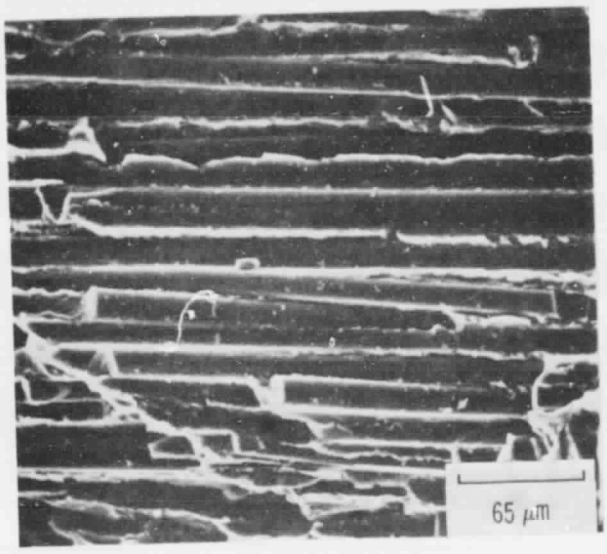
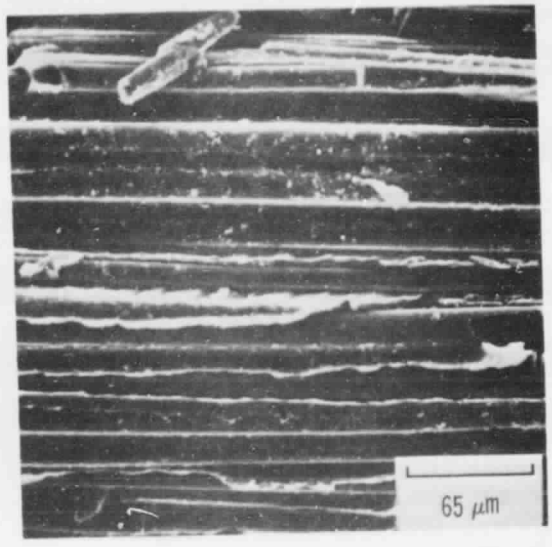


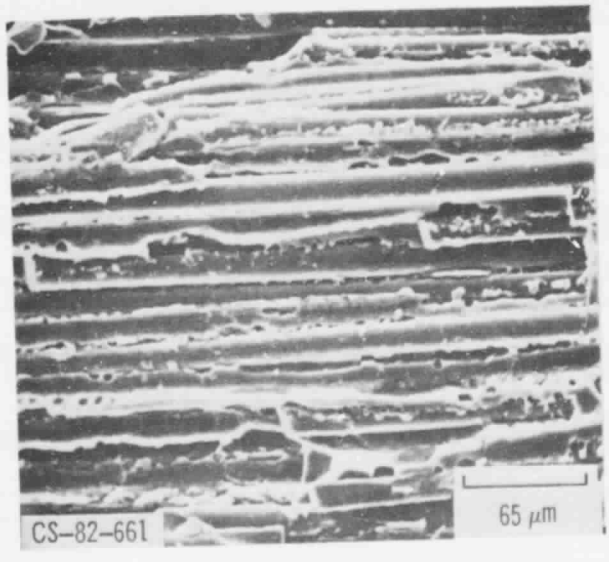
Figure 9. - Diamond pyramid hardness of the matrix in 55 volume percent FP-Al_2O_3/EZ33Mg composites after cycling to 350° C for indicated number of cycles. Matrix hardness values of similar composites isothermally heated at 350° C for up to 150 hours are also shown. (Matrix hardness values of untreated composites are shown at zero cycles.)



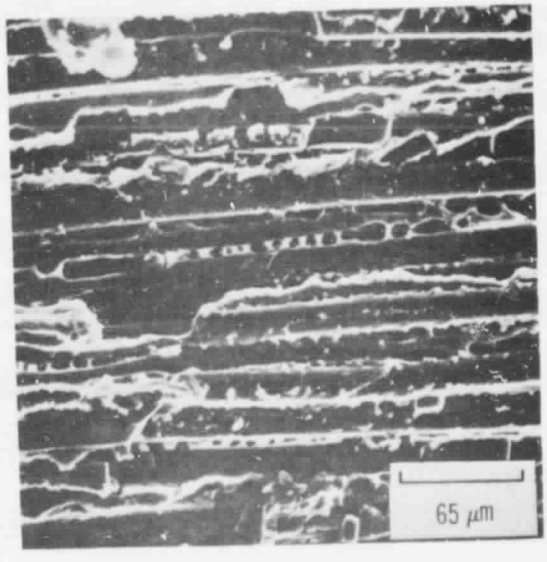
(a) Untreated



(b) 20 Cycles to 350° C



(c) 1000 Cycles to 350° C



(d) 2000 Cycles to 350° C

Figure 10. - Photomicrograph of fracture surface untreated and thermally cycled Fp-Al_2O_3/EZ33Mg composites stressed in transverse direction showing effect of cycling to 350° C for indicated number of cycles.